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Correlations Between Plasma Variables and the Deposition Process of Si Films from Chlorosilanes in Low Pressure rf Plasmas of Argon and Hydrogen

R. Avni
*Lewis Research Center
Cleveland, Ohio*

and

U. Carmi
*Nuclear Research Center
Beer Sheva, Israel*

and

A. Grill, R. Manory, and E. Grossman
*Ben Gurion University of the Negev
Beer Sheva, Israel*

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CORRELATIONS BETWEEN PLASMA VARIABLES AND THE DEPOSITION PROCESS OF Si FILMS
FROM CHLOROSILANES IN LOW PRESSURE rf PLASMAS OF ARGON AND HYDROGEN¹

R. Avni²

National Aeronautics and Space Administration
Lewis Research Center
Cleveland, Ohio 44135

and

U. Carmi

Nuclear Research Center Negev
Beer Sheva, Israel

and

A. Grill, R. Manory, and E. Grossman

Ben Gurion University of the Negev
Beer Sheva, Israel

ABSTRACT

The dissociation of chlorosilanes to silicon and its deposition on a solid substrate in a rf plasma of mixtures of argon and hydrogen were investigated as a function of the macrovariables of the plasma. The dissociation mechanism of chlorosilanes and HCl as well as the formation of Si in the plasma state were studied by sampling the plasma with a quadrupole mass spectrometer. Macrovariables such as pressure, net rf power input and locations in the plasma reactor strongly influence the kinetics of dissociation.

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²Ben Gurion University of the Negev, Beer Sheva, Israel and NRC - NASA Research Associate. Present address: NASA Lewis Research Center, M. S. 23-2, Cleveland, Ohio 44135.

The deposition process of microcrystalline silicon films and its chlorine contamination were correlated to the dissociation mechanism of chlorosilanes and HCl.

INTRODUCTION

The dissociation of the chlorosilane monomers (SiCl_4 , SiHCl_3 , etc.) and the formation of polymerized chlorosilicons and silicon taking place in low pressure (>1.0 torr) argon rf plasma have been shown, in previous publications (refs. 1 to 3), to be governed by an ion-molecule interaction mechanism. Additions of hydrogen in the rf discharge in concentrations above 10 vol % of the gas feed ($\text{SiCl}_4 + \text{H}_2 + \text{Ar}$) enhances both the monomer dissociation and the chlorosilicon polymers formation and inhibits the ion-molecule mechanism towards an excited radical-molecular interaction mechanism (refs. 1 to 3). The criterion discerning which one of the two mechanisms are rate determining is the following: Free radicals, positive and negative ions are continuously formed in the rf plasma as a result of the interaction of the gas monomer molecules with plasmas energetic electrons. The concentration per unit volume of plasma (cm^{-3}) of free radicals, n_R , positive ions, n_i , and negative ions, n_N , and their spatial gradients are variables entering into the kinetics of reactions with the monomer molecules. Another variable controlling the kinetics is the reactivity of the radicals and the charged ions, i.e., the cross sections, σ_R , σ_i , and σ_N of such reactions. The probability of reaction, P , is defined as the product of $n \times \sigma$ in a given location Z , along the plasma system. Thus if

$$n_R \times \sigma_R > n_i \times \sigma_i, \text{ then } P_R > P_i$$

meaning that the probability of radicals interacting with the monomer is higher than the probability for ion-monomer interactions at Z and vice versa

$$P_i > P_R \text{ when } n_i \times \sigma_i > n_R \times \sigma_R \text{ at } Z.$$

The homogeneous reaction kinetics in the $\text{SiCl}_4 + \text{H}_2 + \text{Ar}$ rf discharge for the monomer dissociation and for the formation of chlorosilicon polymers and Si have been diagnosed (refs. 1 to 3) by a quadrupole mass spectrometer system, by paramagnetic resonance for the relative concentration of free radicals and by double floating electrostatic probes for plasma density. The results obtained (refs. 1 to 3) clearly show a high probability, P_R , for the radical mechanism as the rate determining step in the $\text{SiCl}_4 + \text{H}_2 + \text{Ar}$ of plasma.

Whereas the radical mechanism governing the homogeneous reaction is completely or partially responsible, for the heterogeneous reaction as well, is still an open question. Heterogeneous reactions are the plasma - surface interactions (for both uncoated and coated surfaces). The end product of the $\text{SiCl}_4 + \text{H}_2 + \text{Ar}$ rf plasma is the deposition of microcrystalline silicon ($\mu\text{C-Si}$) contaminated by chlorine. The aim of the present work is two-fold:

- (1) To correlate between the homogeneous and heterogeneous reactions leading to the deposition of $\mu\text{C-Si}$, and
- (2) To elucidate the mechanism of chlorine interaction with the $\mu\text{C-Si}$ surface.

Correlations and modeling between deposition rate on a solid substrate and the processes taking place in a plasma have been reported by several investigators. For hydrocarbon plasmas, Yasuda (ref. 4) developed a model for plasma polymerization and polymer deposition based upon reactive species (positive and/or negative ions, or free radicals or excited molecules) forming polymer intermediates in the vapor phase, terminating with a polymer in the solid phase. Denaro et al. (ref. 5) developed a theoretical model to explain the kinetics of styrene polymerization in a 2 MHz discharge. The energy transferred to the polymer film by inelastic electron collisions was assumed

to produce free radicals which were postulated to react with the surface adsorbed monomer and surface adsorbed radicals. Poll et al. (ref. 6) accounted for the effects of the gas phase composition on the rate of polymer deposition. Analog computer predictions of this model have been shown to be in qualitative agreement with the experimental observations (ref. 6). Lam et al. (ref. 7) proposed the initiation of monomers by electron impact, to form free radicals, followed by propagation in which free radicals react with the monomer and the termination step in which the end polymer is formed. The authors (ref. 7) concluded that the model gas phase initiation, surface propagation and termination describe best the polymerization kinetics of styrene. Bell (ref. 8) summarized the various models based upon the free radicals interactions in the plasma polymerization processes of hydrocarbons.

For the polymerization of hydrocarbons, Khait et al. (ref. 9) developed a model based upon positive ions-molecule interactions in the gas phase (ref. 10) and in the plasma layer adjacent to the solid surface by which predictions for deposition rates of hydrocarbon were in agreement with experimental observations at various pressures and rf input powers.

In the silane glow-discharge plasma, Turban et al. (ref. 11) correlated the deposition of a Si:H to the plasma process by four principal steps: Decomposition of SiH_4 generating primary active species, recombination of active species (ions, radicals, atoms), followed by surface reactions between radicals atoms and ions and finally plasma etching and desorption of silane and hydrogen.

The interactions between Si surface and various etchants in low pressure plasmas have been summarized by Flamm (ref. 12), Coburn (ref. 13), Winters et al. (ref. 14), and VINOGRADOV et al. (ref. 15).

The chlorine contamination of the $\mu\text{C-Si}$ films in glow discharges and rf plasmas of chlorosilanes gas mixtures with H_2 and Ar is a well-established

fact (refs. 16 to 21). In the present work combining mass spectrometric (QMS) studies with the values of chlorine contamination as measured by scanning electron microscope with an energy dispersive attachment (EDAX) the mechanism of chlorine interacting with the $\mu\text{C-Si}$ surface was derived.

EXPERIMENTAL

The experimental setups for both the dissociation of SiCl_4 in the plasma state and the deposition of $\mu\text{C-Si}$ have been described elsewhere (refs. 18 to 21). Several features of the experimental methods have to be emphasized.

(1) The deposition experiments were performed in a low pressure (1 to 10 torr) rf plasma (27.12 MHz) with the following mixture of gases: 5 vol % SiCl_4 (Merck) + 15 vol % H_2 (Matheson UHP Grade) + 80 vol % Ar (Matheson UHP Grade). The flow of the gas mixture was kept constant at 70 to 80 SCCM. The deposition time was kept constant to 1 hr. The graphite substrates (ATJ) were located separately in 3 different locations with regard to rf coil and the gas stream, namely:

- (a) position H, upstream at the beginning of the rf coil.
- (b) position G in the center of the rf coil.
- (c) position F, downstream at 1 cm outside the end of the rf coil.

(2) The QMS worked at a pressure lower than 10^{-6} torr and the differential pressure chamber was evacuated to 10^{-5} torr. The orifice between the microwave plasma and the differential pumping is 0.2 mm in diameter whereas the orifice to the ionization chamber of the QMS is 0.3 mm in diameter. The molecular beam reaching the ionization chamber has a sonic velocity with low temperature.

(3) The electron energy in the ionization chamber of the QMS has been reduced to 30 eV in order to reduce fragmentation of polymerized chlorosilicons formed in the plasma.

(4) From the mass spectra results, given as a normalized concentration (I/I_0), the blank spectrum (no plasma) was subtracted, thus enabling the analysis of the influence obtained on the whole spectrum due to a change in any plasma parameter and eliminating the effect of the secondary ionization in the ionization chamber of the QMS.

The SEM, JEOL, JSM35 was used to measure the thickness (d) of the Si deposits. Under the conditions described elsewhere (refs. 19 and 21) the Si films have an 80 percent degree of crystallinity as directly obtained in the rf plasma reactor and microcrystalline structure of $\sim 130 \text{ \AA}$. The EDAX attachment measured the concentration of chlorine in the $\mu\text{C-Si}$ films.

RESULTS AND DISCUSSIONS

Location of Sampling and Deposition

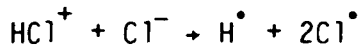
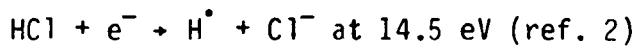
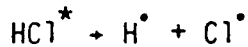
The behavior of the normalized concentration, I/I_0 of chlorosilicon species HCl and Si as measured by the quadrupole mass spectrometer (QMS) in the plasma in positions H, G, and F are shown in Fig. 1(a). In the argon-hydrogen microwave plasma the dissociation of SiCl_4 starts at H and continues sharply its dissociation towards G, the center of the microwave cavity where the maximum energy is delivered to the gas. The dissociation of SiCl_4 in the gas phase results mainly with polymerized chlorosilicon species (up to Si_6), hydrochloric acid and silicon. Besides silicon which has a maximum of its formation at G the other species shown increase their concentration from positions H to F. The deposition of $\mu\text{C-Si}$ for any input rf power reaches a maximum in position G as shown in Fig. 1(b). The results of Si deposition (fig. 1(b)) are similar to the behavior of Si (normalized concentration) as shown in Fig. 1(a) at 2 torr and 100 W, thus the Si deposition on the solid substrate is proportional to its formation in the plasma state.

In Fig. 2 the behavior of the normalized concentration, I/I_0 of chlorosilicon species in the 5 vol % SiCl_4 + 15 vol % H_2 + 80 vol % Ar microwave

plasma versus sampling positions, H, G, and F at 200 W is shown. Comparing with the same mass spectra at 100 W (fig. 1(a)) and considering the changes from position G towards F it was found that:

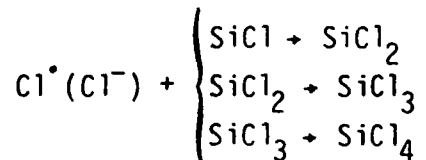
- (1) The normalized concentration of HCl^+ decreases sharply.
- (2) The normalized concentration of SiCl_3^+ and SiCl_4^+ increased sharply.

Position G, the center of the rf coil is where the maximum energy density is delivered to the plasma. Starting at this position and at 200 W input, dissociation of HCl takes place along the stream. The bond dissociation energy of HCl molecule is 4.4 eV (ref. 2), and of HCl^+ is 4.7 eV (ref. 2). The favorable mechanism for the HCl dissociation at low energies is the one resulting in the formation of H^\bullet and Cl^\bullet radicals or of the negative chlorine ion (ref. 22):

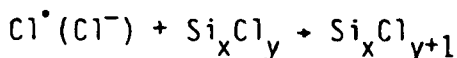


This mechanism probably occurs at lower input powers too, but with a smaller yield.

From position G towards F, the chlorine radicals or the negative ions react with the chlorosilicon species and silicon in a recombination process, resulting in the high concentrations of SiCl_3^+ and SiCl_4^+ (fig. 2(a)). From this behavior it may be assumed that the recombination mechanism yielding SiCl_3 and SiCl_4 takes the form (ref. 22):



or generally:



This recombination process hinders the increase in the normalized concentration of Si.

The behavior of chlorine concentration in the μC Si films as measured by EDAX for the substrate in locations H, G, and F is shown in Fig. 2(b). The chlorine concentration increases continuously from H to F and increases as well with increasing the net rf power input. Upstream in position H, where the gas mixture enters the plasma, the dissociation of HCl is negligible. (fig. 2(a)); the corresponding C_{Cl} in the μC Si films is small, around 1 percent. In position G at a given power input where the maximum energy density was delivered, the dissociation of HCl increases, more chlorine radicals and ions were formed thus increasing the C_{Cl} in the silicon deposit. Downstream, in position F, the dissociation of HCl increases, being a kinetic process (ref. 22), as shown in Fig. 2(a). For a higher dissociation of HCl, a higher chlorine contamination was measured in position F as indicated in Fig. 2(b).

The Net Power Input

The behavior of $I/\Sigma I$ with the net input power in the microwave plasma is shown in Fig. 3(a), for position H. Increasing the input power results with a higher dissociation of SiCl_4 in the microwave plasma thus increasing the formation of HCl, polymerized chlorosilicons (represented by Si_5Cl_3^+) and silicon. In other words, in position H the normalized concentration of HCl and Si is proportional to the power input. The deposition of Si reaches a maximum thickness value at a rf power input of 250 W in position H, decreasing at higher rf power as shown in Fig. 3(b) due to an increase in the etching process as explained below.

The chlorine concentration behavior with the net rf power input in the deposition process of μC Si is shown in Fig. 4. Generally, the C_{Cl}

values increases with increasing power in any of the three locations thus corresponding to the dissociation process of HCl in the plasma state (fig. 2(a)). In Fig. 4 is also shown the behavior of the thickness of $\mu\text{C Si}$ in position F versus the rf power input. At 400 W the $\mu\text{C Si}$ thickness values reached the minimum, whereas the C_{Cl} value the maximum. The minimum "d" values are correlated to an etching process due to chlorine atoms or radicals as follows:

The graphite substrate on which μSi was deposited, was grounded and therefore a luminous zone was formed during the rf discharge in $\text{SiCl}_4 + \text{H}_2 + \text{Ar}$. This luminous zone is formed from a sheath and a plasma layer (ref. 9) around the substrate. Positive ions from the plasma, Ar^+ among others are accelerated by the strong electric fields in the sheaths and energetic ionic flux strikes the solid surface producing short lived hot spots (ref. 9). Thus an ion enhanced chemical etching takes place (refs. 12 to 15). Now, if the etching is responsible for the small thickness values of the $\mu\text{C Si}$ films at high rf powers input, in the F location, then how can the high values of chlorine contamination C_{Cl} in the film be explained? Chuang (ref. 22) after exposing a single crystal Si surface to XeF_2 using XPS technique detected a stable " SiF_2 - like" surface species. It should be remembered then in our deposition process, Si films have microcrystalline nature, i.e., with ample dangling bonds. As not in the case of Si single crystal with no dangling bonds or of polycrystalline Si (crystallite size $> 30 \mu\text{m}$) with few dangling bonds, the microcrystalline Si has enough vacancies to hold chlorine atoms (refs. 16 and 17).

In the tetrachlorosilane plus hydrogen plus argon rf discharge plasma, between deposition and etching an equilibrium is reached which is dependent on the rf input power and location. In the range of 100 to 150 W input rf power the deposition rate reaches its maximum for the substrate in locations H or G

and a minimum chlorine contamination only in location H. In locations G and F at rf power above 200 W the etching rate was increased.

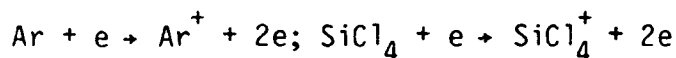
Total Gas Pressure

In the gas phase the behavior of the normalized concentration $I/\sum I$ of SiCl_4 , chlorosilicon species, Si and HCl are shown in Fig. 5(a) for position H as a function of total gas pressure. The dissociation of SiCl_4 with increasing pressure in the microwave plasma results with the formation of polymerized chlorosilicones, hydrochloric acid and silicon. At a pressure of about 3 torr the formation of Si in the gas phase reaches a maximum which remains constant at higher pressures. The maximum thickness d , of the Si films deposited on the solid substrate located in position H, was obtained between 1 to 3 torr, as shown in Fig. 5(b). Comparing the results in Figs. 5(a) and (b), the formation of Si in the gaseous state is not proportional with the deposition of Si on the substrate, for pressures above 3 torr.

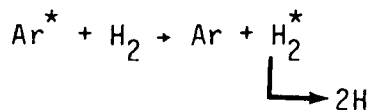
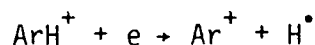
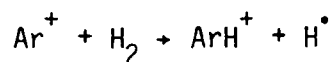
The Dissociation and the Deposition Mechanism

The dissociation mechanism and kinetics of SiCl_4 to Si and HCl by Ar and H_2 in the gaseous state of the microwave plasma has been reported elsewhere (ref. 1) and follows the following pattern:

(1) Initiation - fragmentation and formation of chlorosilane radicals and positive ions:

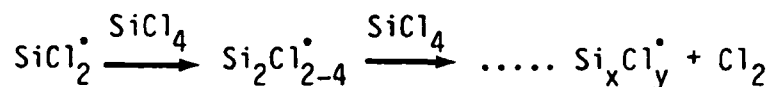


$\text{Ar}^+ + \text{SiCl}_4 \rightarrow \text{SiCl}_3^+ + \text{Cl} + \text{Ar}$ and/or by Penning ionization. The initiation of radicals is mainly due to H_2 in the microwave plasma by:



Followed by $H^\bullet + SiCl_4 \rightarrow SiCl_3^\bullet + HCl$

(2) Propagation – formation of polymerized silicon species by radicals-molecule interactions:



(3) Termination – formation of Si and HCl.

In the plasma state at higher input power and higher pressures dissociation and recombination processes occur, as reported. The recombination process hinders the increase of Si concentration in the gas phase, and the formation of $\mu C-Si$ films.

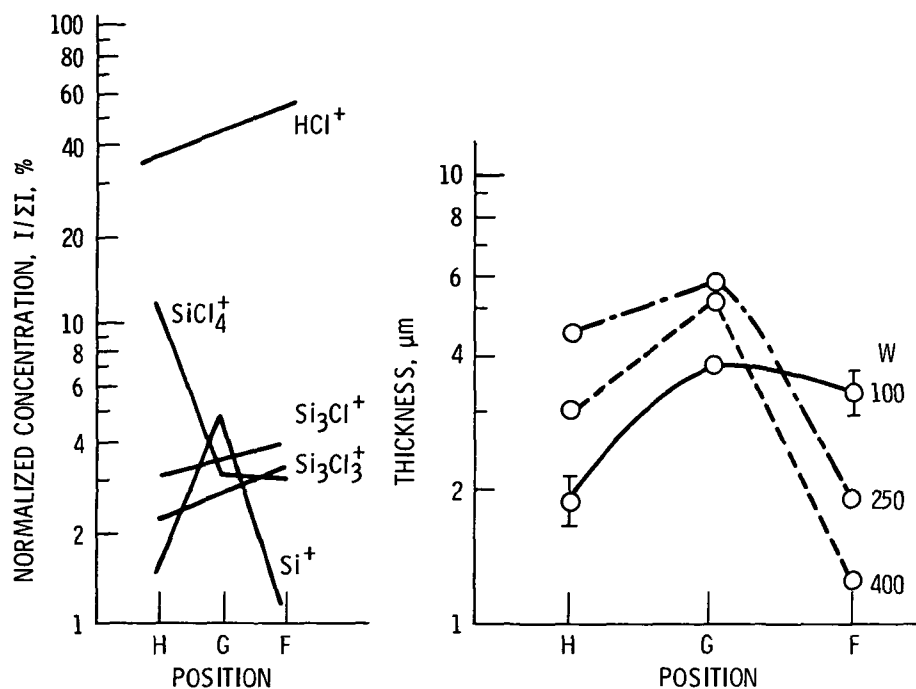
CONCLUSION

It has been shown the direct influence of the mechanisms and kinetics of radical molecule homogeneous interactions (in the plasma state) on the heterogeneous reactions on $\mu C-Si$ in its deposition etching and chlorine contamination phenomena.

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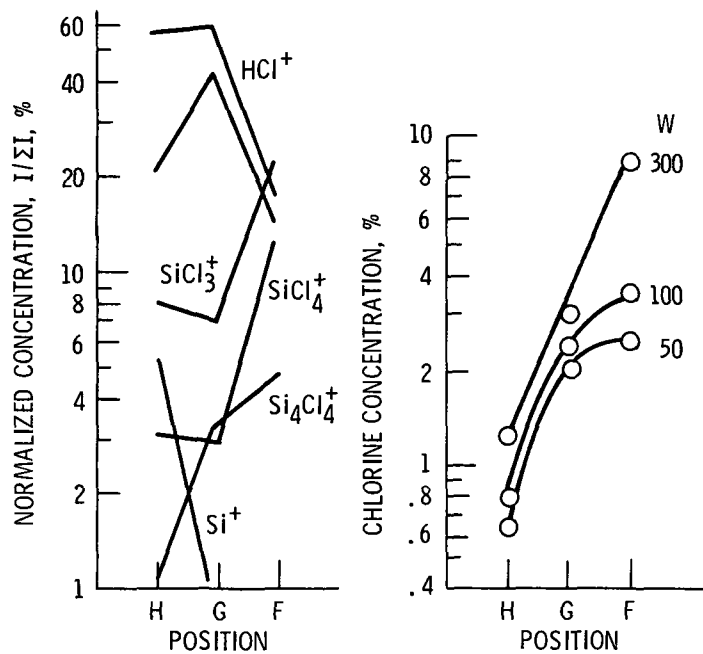
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(a) Normalized concentrations of several mass species at different locations in the plasma reactor. Pressure = 2 torr; power = 100 W; ionization energy = 30 eV; gas mixture = 5 v/o SiCl_4 + 15 v/o H_2 + Ar.

(b) Thickness of Si films obtained versus substrate position in the plasma reactor. Pressure = 2 torr; time = 1 hr; gas mixtures, as in Fig. 1a.

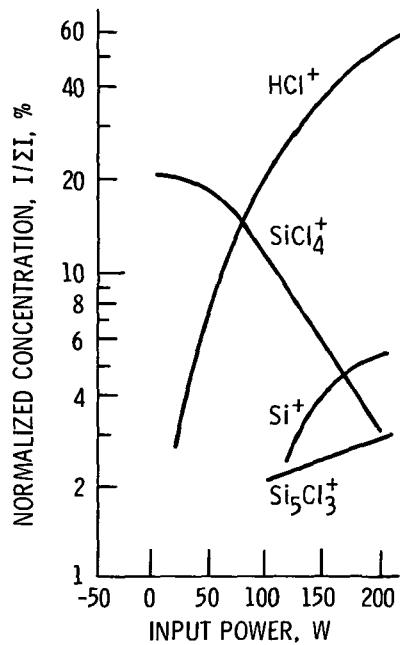
Fig. 1. - Reactions in the plasma and deposition of Si films versus position.



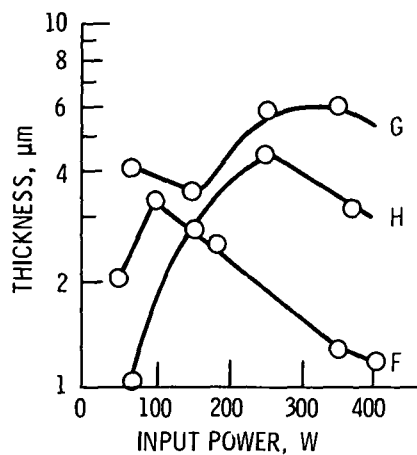
(a) Normalized concentrations at different locations in the plasma reactor. Pressure = 2 torr; power = 200 W; ionization energy, 30 eV; gas mixtures as in Fig. 1a.

(b) Chlorine concentration in Si films versus substrate locations in the plasma reactor. Conditions as in Fig. 1b.

Fig. 2. - Reactions in the plasma and chlorine concentration in Si films versus position.



(a) Normalized concentrations of several mass species versus input power at position H in the plasma reactor. Conditions as in Fig. 1a.



(b) Thickness of Si films at different locations versus input power. Conditions as in Fig. 1b.

Fig. 3. - Reactions in the plasma and deposition of Si films versus input power.

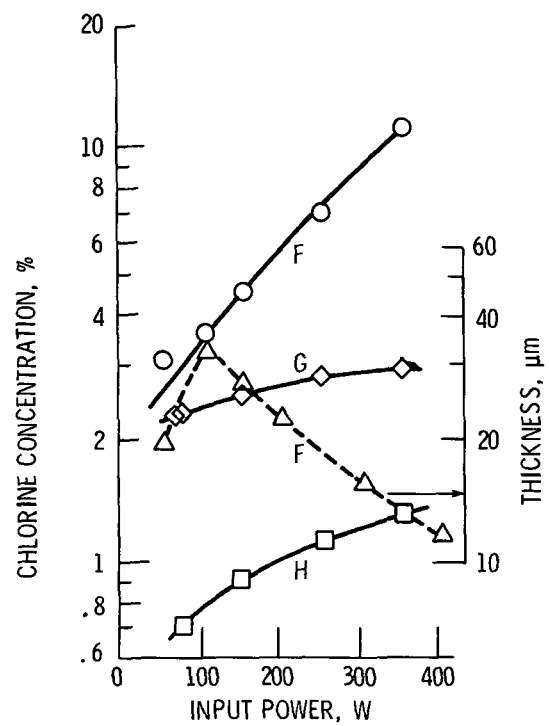
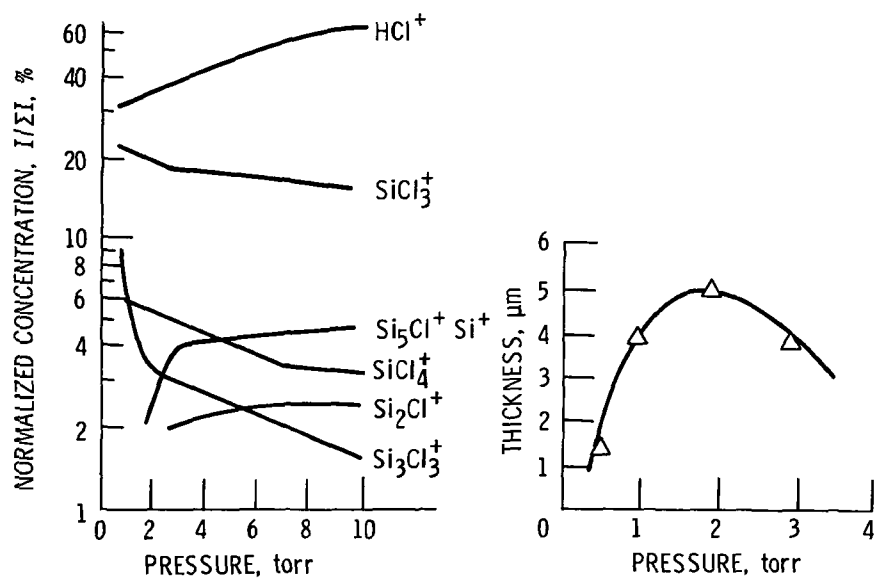


Fig. 4. - Chlorine concentration in Si films and thickness (location F) versus input power. Conditions as in Fig. 1b. (--- Si thickness.)



(a) Normalized concentration of several mass species versus gas pressure at position H and 100 W. Conditions as in Fig. 1a.

(b) Thickness of Si films versus gas pressure at position H and input power of 150 W.

Fig. 5. - Reactions in the plasma and deposition of Si films versus pressure.

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